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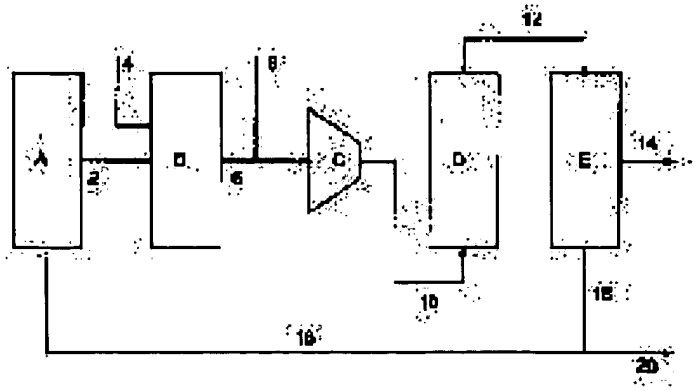
(54) METHOD FOR SEPARATING AND REMOVING PERFLUOROCARBONS FROM GAS STREAM

(57)Abstract:
PURPOSE: To provide an effective method for removing perfluorocarbons from a gas stream by adsorption.
CONSTITUTION: Prefluorocarbons are recovered from a gas stream by supplying the gas stream to an adsorption process using one or two or more energetically uniform adsorbent beds such as a high silicone adsorbent to FAU structure, a high silicone adsorbent of BEA structure and a high silicone adsorbent of MOR structure. As the adsorption process, a pressure swing adsorption or a temp. swing adsorption is preferable.



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CLAIMS

[Claim(s)]
[Claim 1]It is the method of separating said at least one sort of gaseous perfluoro-carbonizing-ized hydrogen from a gas flow containing perfluoro-carbonizing-ized hydrogen, one sort, or two sorts or more of permanent gas, Said gas flow High silicon adsorbent of FAU structure, high silicon adsorbent of BEA structure, High silicon adsorbent of MOR structure, a carbon molecular sieve which has a diameter of a hole of 4.5 Angstrom units at least, How to adsorb said at least one sort of perfluoro-carbonizing-ized hydrogen from said gas flow by through and it at adsorbent chosen from a carbonized sulfonation styrene divinylbenzene copolymer, nature silicate of a middle hole of M41 S-structure class, and these mixtures.
[Claim 2]A way according to claim 1 said at least one sort of gaseous perfluoro-carbonizing-ized hydrogen has 1-8 carbon atoms.
[Claim 3]A method according to claim 2 by which said at least one sort of gaseous perfluoro-carbonizing-ized hydrogen is chosen from tetrafluoromethane, hexafluoroethane, oct fluoropropane, tetrafluoroethylenes, and these mixtures.
[Claim 4]A method according to claim 1 by which said one sort or two sorts or more of permanent gas is chosen from nitrogen, oxygen, argon, and these mixtures.
[Claim 5]A method according to claim 3 by which said one sort or two sorts or more of permanent gas is chosen from nitrogen, oxygen, argon, and these mixtures.
[Claim 6]A way according to claim 1 said adsorbent is high silicon adsorbent of FAU structure.
[Claim 7]A way according to claim 6 said adsorbent is dealuminization type Y type zeolite.
[Claim 8]A way according to claim 7 said dealuminization type Y type zeolite has silicon versus an aluminum ratio of at least 100 [about].
[Claim 9]From a gas flow containing nitrogen, are one sort or two sorts or more of gaseous perfluoro-carbon a circle method adsorption method for dissociating, and the :(a) aforementioned gas flow, High silicon adsorbent of FAU structure, high silicon adsorbent of BEA structure, high silicon adsorbent of MOR structure, A carbon molecular sieve which has a diameter of a hole of 4.5 Angstrom units at least, A sulfonation styrene divinylbenzene copolymer, nature silicate of a middle hole of M41 S-structure class which were carbonized, To at least one adsorbent bed chosen from these mixtures, and through, How to consist of desorbing one sort or two sorts or more of gaseous perfluoro-carbon which stuck to said one sort or two sorts or more of gaseous perfluoro-carbon from said gas flow and in which (b)

adsorption was subsequently carried out by it from said adsorbent.
[Claim 10]A method according to claim 9 by which said circle method adsorption method is chosen from a pressure-swing-adsorption method, temperature swing adsorption processes, and such combination.
[Claim 11]A method according to claim 9 by which an adsorption process of said circle method adsorption method is performed with abbreviation-100 - temperature of the range of 100 ** of abbreviation, and absolute pressure of the range of about 0.5-20 bars.
[Claim 12]A method according to claim 9 by which said one sort or two sorts or more of gaseous perfluoro-carbon is chosen from tetrafluoromethane, hexafluoroethane, oct fluoropropane, tetrafluoroethylenes, and these mixtures.
[Claim 13]A way according to claim 12 said adsorbent is dealuminization type Y type zeolite.
[Claim 14]A way according to claim 13 said dealuminization type Y type zeolite has silicon versus an aluminum ratio of at least 100 [about].
[Claim 15]A gas mixture thing which is how to defecate ** containing a chemical residue by silicon steam treatment, and contains a resultant, oxygen, and unreacted perfluoro-carbon for perfluoro-carbon and oxygen of :(a) gas by through and it in ** is formed.;
(b) Introduce inactive gas chosen from nitrogen, argon, and these mixtures into said gas mixture thing.;
A compound which reacts said gas mixture thing to said resultant in an included reactor (c) Through, It removes said resultant from said gas mixture thing substantially, However, a gas mixture thing which does not include a resultant substantially [above /; and / (d)] as does not have remarkable influence on said perfluoro-carbon in this case, High silicon adsorbent of FAU structure, high silicon adsorbent of BEA structure, high silicon adsorbent of MOR structure, A carbon molecular sieve which has a diameter of a hole of 4.5 Angstrom units at least, A sulfonation styrene divinylbenzene copolymer, nature silicate of a middle hole of M41 S-structure class which were carbonized, and; which separates said perfluoro-carbon from a gas mixture thing in which an adsorption process of a circle method using at least one adsorbent bed chosen from these mixtures is presented, and the above does not include a resultant substantially by it -- a method including these each process.
[Claim 16]A method according to claim 15 of including further making the aforementioned room recycle separated perfluoro-carbon.
[Claim 17]A method according to claim 15 of including further carrying out scrubbing of said gas mixture thing using a solvent for at least one sort of resultants included in said gas mixture thing in advance of a process (c).
[Claim 18]A way according to claim 15 the aforementioned room is a deposition chamber or a silicon chip etching chamber.
[Claim 19]A way according to claim 15 said adsorbent is dealuminization type Y type zeolite.
[Claim 20]A way according to claim 19 said dealuminization type Y type zeolite has silicon versus an aluminum ratio of at least 100 [about].
[Claim 21]It is how to polymerize tetrafluoroethylene, :(a) tetrafluoroethylene is contacted for a catalyst in a polymerization reaction machine, and a generation mixture which contains polytetrafluoroethylene and unreacted tetrafluoroethylene by it is formed.;
(b) Strip said generation mixture using inactive gas, form a gas mixture thing which contains

stripping gas and unreacted tetrafluoroethylene by it and by which stripping was carried out, and a gas mixture thing of; and (c) above by which stripping was carried out, High silicon adsorbent of FAU structure, high silicon adsorbent of BEA structure, high silicon adsorbent of MOR structure, A carbon molecular sieve which has a diameter of a hole of 4.5 Angstrom units at least, A sulfonation styrene divinylbenzene copolymer, nature silicate of a middle hole of M41 S-structure class which were carbonized, and; which presents an adsorption process of a circle method using at least one adsorbent bed chosen from these mixtures, and separates unreacted tetrafluoroethylene from a gas mixture thing in which stripping of the above was carried out by it -- a method including these each process.

[Claim 22]A method according to claim 21 of including further making said polymerization reaction machine recycle unreacted tetrafluoroethylene from a process (c).

[Claim 23]A way according to claim 21 said adsorbent is dealuminization type Y type zeolite.

[Claim 24]A way according to claim 23 said dealuminization type Y type zeolite has silicon versus an aluminum ratio of at least 100 [about].

[Claim 25]A method according to claim 21 by which said inactive gas is chosen from nitrogen, argon, and these mixtures.

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DETAILED DESCRIPTION

[Detailed Description of the Invention]
[0001]
[Industrial Application]Especially this invention relates to the way adsorption removes perfluoro-carbonizing-ized hydrogen from a gas flow, about purification of a gas flow.
[0002]
[Background of the Invention]The hydrocarbon derivative (perfluoro-carbon) fully fluoro-ized (fluorination) is globally used in various home use and the industrial use field. Gaseous perfluoro-carbonizing-ized hydrogen is emitted into environment now as a result of such use. As an example, the perfluoro-carbon of low molecular weight is used combining oxygen in the semiconductor manufacture for etching of a silicon chip, and purification of a chemical-vacuum-deposition room. These processes are typically performed under a vacuum. The exhaust gas of ****** includes various resultants, such as the others and unreacted vacuum evaporation compound and hydrogen fluoride of perfluoro-carbon, and nitrogen trifluoride. Since these compounds cannot be emitted safely in the atmosphere, exhaust gas is processed in order to change it to the compound which generally destroys the compound considered to be harmful, or can emit them into the atmosphere. According to one procedure, a gas flow is introduced into a reactor (manufacturer: Edwards High Vacuum International Division of BOC Group (trade name: EDWARDS glass fiber reinforced cement)), for example, a gas reaction tower, The ingredient of a gas flow reacts at an elevated temperature in it, and it is changed into destroyable solid matter. However, perfluoro-carbon is nonresponsiveness highly and it passes a reactor, without being influenced. Since it is not thought to the ozone layer which perfluoro-carbon is avirulence and surround the earth that it is harmful, they are emitted into the atmosphere now.
[0003]However, it is thought that perfluoro-carbon is what carries out warming of the earth on account of those advanced stability and the thermal characteristic (global warmers). or [therefore, / that the industrial world in the world minimum-izes discharge of the perfluoro-carbonizing-ized hydrogen to the inside of environment now] -- or the efforts for for stopping are performed. The process available now although these compounds are collected from abandonment gas is considered in how to destroy [expense increases, and] them since it is not necessarily practical. In order to destroy, one proposed method is combustion. This is performed by heating them in temperature of not less than 1000 **, and hydrogen and

oxygen can be given to the temperature by burning under existence of perfluoro-carbon.
[0004]
[Problem(s) to be Solved by the Invention]Destruction of perfluoro-carbon is not the best solution over the problem of abandonment. That is because expense increases and other harmful by-products arise by incomplete combustion. Perfluoro-carbon is output with high value, and if it can collect them from a gas flow at the expense which is not high, it will be beneficially recyclable. This invention provides a method effective in expense, and efficient, in order to attain this purpose.
[0005]
[Means for Solving the Problem]According to the large mode of this invention, a gas flow, By letting it pass to adsorbent (mesoporous) of micro-cellular adsorbent of one sort or two sorts or more of specific high silicon (high silicon) homogeneous in energy and/or one sort, or two sorts or more of specific qualities of a middle hole homogeneous in energy, One sort or two sorts or more of gaseous perfluoro-carbon is separated from said gas flow containing at least one sort of permanent gas. Perfluoro-carbon is adsorbed more strongly than an ingredient of others of a gas flow by these adsorbent. Perfluoro-carbon is recovered from adsorbent by the usual reproduction procedure.
[0006]Desirable adsorbent is dealuminization type Y type zeolite, dealuminization type beta type zeolite, and dealuminization type mordenite (mordenite), and silicon versus an aluminum ratio is 50 or more about these all. The most desirable adsorbent is Y type zeolite of a 100 or more dealuminization type [aluminum ratio / silicon versus].
[0007]A method of this invention can be used for collecting all the perfluoro-carbon of a state of a gas or a steam at adsorption temperature. Especially this invention is suitable for recovery of saturation or ethylene unsaturation perfluoro-carbon, It is suitable for recovery of the perfluoro-carbon, for example, perfluoro-methane, which has especially a carbon atom to eight pieces, perfluoro-ethane, perfluoro-ethylene, perfluoro-hexane, perfluoro-octane, etc.
[0008]Generally specifying an adsorptive process used can use all adsorption procedures rather than it is important in this invention. Generally an adsorptive process of a circle method is used and cycles of a pressure-swing-adsorption method (PSA) and a temperature swing adsorption process (TSA) or such combination are preferred. As for adsorption, in two or three beds or more which became the lot arranged in parallel, it is preferred to be operated by non-aligning formula so that another floor may be reproduced, when adsorption is presented with at least one floor.
[0009]In addition to a mere adsorptive process which was mentioned above, it can use for this invention changing a specific circulating treatment process. Vacuum deposition, an etching chamber purification process, and a perfluoro-ethylene polymerization-ized process are one of specific circulating treatment processes which can incorporate this invention.
[0010]In vacuum evaporation and purification of an etching chamber, ** containing a vacuum evaporation thing or an etching chemical-vacuum-deposition thing is defecated by introducing perfluoro-carbon and oxygen under a plasma condition into **. Perfluoro-carbon and oxygen react to a vacuum evaporation thing, and form various gas products. A resultant, an unreacted vacuum evaporation chemical, perfluoro-carbon, and a gas mixture thing that consists of oxygen are taken out from a deposition chamber (preferably exhaust air), and is arbitrarily

diluted with nitrogen or argon, and, subsequently to a reactor, it lets it pass at an elevated temperature. A vacuum evaporation chemical changes with them to a harmless solid. Although perfluoro-carbon does not change within a reactor, it is picked out from a gas reaction tower distillate by above-mentioned adsorptive process, and is recycled to a deposition chamber or an etching chamber, or is stored for future use. Perfluoro-carbon can be given to further purifying process like condensation or low temperature distillation, when the purity needs to be raised.

[0011]In a polymerization of perfluoro-ethylene, perfluoro-ethylene polymerizes in a reactor and a mixed output of polymer and an unreacted monomer arises by it. An unreacted monomer is preferably removed from a mixed output by stripping using inactive gas. Subsequently, a monomer by which stripping was carried out is given to an above-mentioned adsorption process, and is adsorbed by it in perfluoro-ethylene. After being desorbed from a bed, perfluoro-ethylene is recycled to a polymerization reaction machine, or is sent to a storage area, or it lays on the shelf of it.

[0012]

[A desirable mode for inventing] Perfluoro-carbon is separated from one or two permanent gas or more by a method of this invention. Nitrogen, oxygen, argon, helium, neon, krypton, a xenon, hydrogen, and carbon monoxide are contained in permanent gas on the purpose of this invention. Especially this invention is suitable for separating perfluoro-carbon from nitrogen, oxygen, argon, and these mixtures.

[0013]It is a specific substance homogeneous in energy, and a substance of microporosity which has a diameter of a hole of about 4.5 Angstrom units at least, and quality of a middle hole is among adsorbent which can be used by this invention. It is defined as a substance which has the hole size of an average of less than about 20 Angstrom units with a micro-cellular substance on the purpose of this explanation, and is defined as a substance which has average hole size in the range of about 20 - about 500 Angstrom units with a substance of quality of a middle hole. In adsorbent homogeneous in energy, adsorption energy of all the absorption positions is substantially equivalent. Even if this definition has substantially constant heat of measurable adsorption experimentally and is a time of concentration of an adsorbed compound changing, it means that that is right. It is used here. "Heat of substantially fixed adsorption" means that heat of adsorption of the above-mentioned substance does not change at not less than about 10%.

[0014]Refining FAU structure class zeolite of high silicon [adsorbent / suitable / micro-cellular], For example, a thing of dealuminization type Y type; Refining BEA structure class zeolite of quantity silicon, For example, dealuminization type beta-type-zeolite; and refining MOR structure class zeolite of high silicon, for example, dealuminization type mordenite, a thing which has a diameter of a hole of about 4.5 Angstrom units at least by a carbon molecular sieve (CMS), ******.

[0015]It is used here. The word "quantity silicon (silicon-rich)" means that silicon versus an aluminum ratio of a molecular sieve is about 50 or more. In the most desirable mode, silicon versus an aluminum ratio of a molecular sieve is about 100:1 or more. A suitable high silicon molecular sieve can be manufactured direct synthesis or by dealuminization-izing a molecular sieve of a desired class. A method of preparing a high silicon molecular sieve and them is

known well, and those structures and manufacturing methods do not constitute any portions of this invention.

[0016]In adsorbent of suitable quality of a middle hole, polymerization coal property adsorbent, For example, sulfonation (it carbonized) styrene divinylbenzene copolymer (for example, product marketed with trademark of Ambersorb from Rohm andHaas); and nature silicate of a middle hole of M41 S-structure class, ****** which carried out the pyrolysis selectively.

[0017]Perfluoro-carbon separable by a process of this invention is usually gaseous things, i.e., in temperature and a pressure by which a gaseous thing or an adsorption process is carried out in ambient air temperature and atmospheric pressure, it is a thing of quality of a steam. The word "perfluoro-carbonizing-ized hydrogen" means an aliphatic hydrocarbon derivative by which all the hydrogen atoms were replaced with a fluorine atom. Saturation and ethylene unsaturation perfluoro-carbon which have the boiling point about 100 ** or less are one of those are contained in a compound of this class, and the perfluoro-carbon which has eight or less carbon atoms is contained in it. As a typical example of perfluoro-carbon recoverable by a process of this invention, there are perfluoro-methane, perfluoro-ethane, perfluoro-propane, perfluoro-hexane, perfluoro-octane, perfluoro-ethylene, etc.

[0018]With reference to an accompanying drawing, it explains so that this invention may be understood well. In a drawing, the same reference mark shows a the same or similar component of a device. The auxiliary device which is not required for an understanding of this invention, for example, a compressor, a heat exchanger, a valve, etc. are excluded from a drawing, in order to give explanation of this invention plain.

[0019]If drawing 1 is referred to, a vacuum deposition chamber of one or a lot or an etching chamber, and C show a vacuum means, D shows a reactor, and, as for A, E shows an adsorber, as for a perfluoro-carbon storage container and B. Composition of these units and details of an operation are known well, and they do not constitute any portions of this invention.

[0020]Perfluoro-carbon and oxygen are supplied to ** B through the lines 2 and 4, respectively, and ** is exhausted through the line 6. The line 6 connects ** B with an entrance of the vacuum means C. The vacuum means C is a vacuum pump typically. The vacuum means C is connected with the reactor D through the line 10 by the end part. The reactor D is provided with the following.

One sort or two sorts or more of substances reacted to an ingredient of a gaseous process stream.

A means for heating this gas reaction tower to desired reaction temperature (not shown).

Details of the gas reaction tower D do not constitute any portions of this invention, therefore they are not clarified in this explanation. The gas reaction tower D is connected with the unit E by the line 12 in the exit end.

[0021]The abandonment gas exhaust line 14 and the perfluoro-carbon exhaust line 16 are established in the unit E. The line 16 is connected with the perfluoro-carbon recirculation line 18 (this is provided in order to return purified perfluoro-carbon to the container A), and the perfluoro-carbon exhaust line 20 in an illustrated example.

[0022]Various gassing units, for example, a filter, or solvent-cleaning scrubbers are arranged

between the units D and E or on the line 14 between the units C and D of a device, and it may be made to remove an ingredient of particulate matter or fusibility from a device by request. However, since it is not important about this invention, they are not illustrated.

[0023]The main purposes of the unit E are to separate perfluoro-carbon from a gaseous effluent from the gas reaction tower D. Typically, the unit E is a pressure-swing-adsorption device or a temperature swing adsorber, and consists of two or more settled floors which was preferably filled up with one sort or two sorts or more of molecular sieve adsorbent of a kind homogeneous in energy mentioned above. Generally a floor is arranged in parallel, and it is adjusted so that it may operate by a cyclic process which consists of adsorption and desorption. Usually, a device with which adsorption is performed consists of two or more beds of a non-aligning formula, and while one or two beds or more are operating in an adsorption stage which is a cycle, one or other two beds or more are reproduced so that it may be carried out.

[0024]When enforcing a method of this invention shown in drawing 1, perfluoro-carbon and oxygen let the lines 2 and 4 pass, respectively, and it is introduced into ** B of a device which chemical vacuum deposition or etching operation just completed. **** -- various chemical abandonment deposition substances are included, and it is necessary to remove those substances from ** so that ** may be prepared for the next chemical vacuum deposition or etching operation Perfluoro-carbon and oxygen contact an abandonment deposition substance, and react to them, and a gaseous abandonment output generates them. A gaseous output is taken out from a deposition chamber by suction produced by the vacuum means C with unreacted perfluoro-carbon and oxygen. Since a gaseous output and unreacted oxygen form a mixture of a flammability, in order to prevent combustion of an early stage of production gas, inactive gas, for example, nitrogen, argon, and carbon dioxide are introduced into the line 6 through the line 8. Subsequently to inside of the reactor D a gas mixture thing which passed through the line 10 is introduced, and a mixture is heated by not less than about 600 ** in it. Various ingredients of a heated mixture contact reacting matter in a tower, and change to output which can be safely discharged into environment or can be easily collected by future chemical preparation. Unreacted perfluoro-carbon in the line 10 passes the unit D without changing. Effluent gas from a gas reaction tower ranks second, goes into the device E, and receives gas adsorption in it.

[0025]An adsorptive process consists of repeating an adsorption process and a regeneration process of a floor. In a desirable mode, an adsorptive process is pressure swing adsorption, temperature swing adsorption, or these two combination, and a specific adsorption method is determined by chemical composition of process gas. Although specific conditions at the time of an adsorptive process being carried out determine efficiency of an adsorptive process, they do not constitute a part of this invention. These conditions are well known to a person skilled in the art of a field of a gas adsorption process, and all combination of an operating condition which is used by an adsorptive process and which changes widely can be used by a method of this invention.

[0026]Generally, an adsorption process is performed from abbreviation-100 ** or less than it with temperature of a range up to about +100 **, and absolute pressure of a range up to about 0.5 to about 20 bars, and is usually preferably performed with temperature of a range

from about 15 ** to about 75 **, and absolute pressure of a range up to about 1 to about 10 bars. Separation performance of adsorbent is so good that temperature is low. Distributed gas is introduced into an adsorber between adsorption processes of a process, and it flows in [each] a floor in an adsorption stage of a cycle. When gas flows in a floor, adsorbent is adsorbed in perfluoro-carbon. When an adsorption process advances, a front of adsorption formed in front end of adsorbed perfluoro-carbon moves forward in the direction of an exit of gas by which it does not adsorb. The remainder of a gas flow passes through a floor and comes out of the device E through the line 14 as abandonment gas. Abandonment gas may be discharged in the atmosphere, supposing it does not contain an ingredient harmful to environment. Or it may be sent to a downstream device for the further processing. When a front of adsorption reaches a position of a request of a bed, a flow of distributed gas to inside of a floor stops. This shows an end of an adsorption stage of a separation process.

[0027]A floor which completed an adsorption stage receives reproduction next. Similarly conditions for reproduction of a floor to be performed are not important, in order to obtain a good result in operation of this invention. Although reproduction of a PSA floor can be performed with absolute pressure of about 100 mb or less, it is usually performed with absolute pressure of the range of about 100 to about 1000 mb. reproduction of a TSA floor is performed by heating adsorbent to a temperature higher than temperature to which an adsorption process is carried out -- the temperature -- typical -- temperature of the range of about 0 to about 200 ** -- it is the temperature of the range of about 20 to about 150 ** preferably. desorption -- a heater -- and/or, it can carry out by letting a steam or heated inactive gas pass in a floor. A pressure between regeneration processes of a TSA cycle and in an adsorption vessel is comparable as a pressure currently maintained within a container between adsorption processes, or can be made lower than it. It is often preferred to perform a temperature swing process in atmospheric pressure or its neighborhood. When combination of a pressure-swing-adsorption cycle and temperature swing adsorption cycles is used, temperature is high and a pressure is lower than a time of a cycle being in an adsorption process between regeneration processes of a floor.

[0028]Perfluoro-carbon is desorbed from the unit E through the line 16 during reproduction. If collected perfluoro-carbon is a thing of purity suitable for it making a device recycle, it can be returned to the storage container A through the line 18. Or it can be discharged from a device through the line 20 for the further purification.

[0029]In a mode of this invention shown in drawing 2, F shows a polymerization reaction machine, G shows a polymer collection unit, and H shows a tetrafluoroethylene adsorber. All these units and devices are known well and concrete details of those composition and an operation do not constitute a part of this invention.

[0030]The monomer supply line 30 and the polymer exhaust line 32 are attached to the reactor F. The reactor F is a tetrafluoroethylene polymerization reactor of a typical batch type or continuous system, It is provided with a means for agitating contents of a reactor, a means for heating contents of a reactor, etc. while all the standard features required for polymerization-izing of a tetrafluoroethylene monomer, for example, a catalyst supply line, and a polymerization are performed, but these neither is illustrated.

[0031]The polymer exhaust line 32 is connected to an entrance of polymer collection unit G.

The unit G is a stripping unit typically and the stripping gas entrance line 34, the polymer recovery line 36, and the outlet line 38 of gas by which stripping was carried out are attached to it. or [that adsorption unit H is similar to the unit E of drawing 1 although the line 38 is connected to an entrance of adsorption unit H] -- or it is the same. The abandonment gas lines 40 and the tetrafluoroethylene recovery line 42 are attached to the unit H.

[0032]When carrying out this invention with the device of drawing 2, an additive agent of a request of tetrafluoroethylene and others, for example, a catalyst, a polymer modifier, etc., is introduced into the reactor F through the line 30 or another supply line. Polymerization-ization can be performed to a batch type or continuous system in the liquid phase or the gaseous phase. A mixture of polymer output and an unreacted monomer is picked out from the reactor F through the line 32, and is introduced into polymer collection unit G. In the unit G, stripping of the polymer is carried out using inactive gas, for example, nitrogen, or argon, and stripping of the unreacted monomer is carried out by it from polymer. Polymer by which stripping was carried out is taken out from the unit G, and it is sent to further handling unit downstream from a device of drawing 2, A gas flow containing stripping gas and unreacted tetrafluoroethylene by which stripping was carried out is discharged from the unit G through the line 38, and, subsequently to inside of adsorption collection unit H, is introduced. In the unit H, unreacted tetrafluoroethylene is the method mentioned above about a device of drawing 1, and is adsorbed from supply flow. Subsequently, adsorbed tetrafluoroethylene is desorbed from adsorbent, when a polymerization process is a thing of continuous system, it is recycled to the reactor F through the line 42, or when a polymerization process is a thing of a batch type, it is sent to a tetrafluoroethylene storage container.

[0033]

[Example]Although the following examples explain this invention further, unless it is shown in particular, a part, percentage, and a ratio are expressed with the capacity standard.

[0034]Example At 1/4 1 inch (6.35 mm) in diameter, to a gas chromatograph tower 2 feet (61 cm) in length. It was filled up with the pellet (this is marketed with the trademark of Degussa Wessalith DAY type zeolite from Degussa AG) with which extrusion molding of the dealuminization type Y type zeolite was carried out. It let helium as carrier gas pass in the tower by the flow of 30 ml/min using the tower maintained by the temperature of 0 **. The sample of the gas mixture thing which consists of nitrogen, 1% of tetrafluoromethane (CF_4), and 2% of hexafluoroethane (C_2F_6) was poured in into carrier gas, and it let it pass in the tower filled up with adsorbent. The elution time of each ingredient is shown in Table 1. The separation factor of CF_4/N_2 about each examination and C_2F_6/N_2 is measured, and these also show in Table 1. The above-mentioned procedure was repeated at the temperature of 20 ** and 30 **. The result of these examinations is also shown in Table 1.

[0035]

[Table 1]

試験No.	吸着温度 (℃)	滞留時間（分）			分離係数	
		N_2	CF_4	C_2F_6	CF_4/N_2	C_2F_6/N_2
1	0	0.67	2.96	—	4.42	—
2	20	0.57	2.10	15.29	3.68	26.83
3	30	0.52	1.58	11.06	3.07	21.48

[0036]Example In 2 this example, a series of pressure-swing-adsorption examinations were done using various nitrogen-hexafluoroethane (C_2F_6) gas mixture things as a feed stock. The supply flow about the examinations 1, 2, 3, and 5 consists of nitrogen and C_2F_6. The supply flow about the examination 4 contains nitrogen and 4% of oxygen other than C_2F_6. All the examinations were done at 20 inches (50.8 cm) in length in the adsorption vessel of the cylindrical shape of 1.25 inches (31.8 mm) in diameter a couple. The bed was filled up with Degussa Wessalith DAY type zeolite in the examinations 1-4, and the floor was filled up with Ambersorb 563 (trademark) adsorbent in the examination 5. The adsorption vessel of each other was operated at a non-aligning ceremony by the adsorption cycles for 30 minutes. This cycle has each process of the equation of the application-of-pressure =3 second; adsorption =447 second; floor of a feed stock, the equation of the exhaust air =447 second; floor of a decompression =3 second; floor, and backfilling (backfill) = 3-second; of recompression =3 second; output. In the equation of the floor, in the examination of 1, 2, and 5, the equation of the floor went for the forward direction to the opposite direction in (top-to-top) and the examination of 3 and 4 (top-to-bottom). It was carried out by the various temperature and flows which adsorption is performed by the pressure of 3.77 bars, and are shown in Table 2. The adsorption vessel was exhausted to the absolute pressure of 100 mb with the vacuum pump between exhaust processes. Adsorption temperature, a supply flow rate (a part for liter/), the concentration of the hexafluoroethane in a distributed gas flow and a product gas flow, and the recovery rate of C_2F_6 are shown in Table 2 (examinations 1-4).

[0037]
[Table 2]

試験 No.	吸着温度 (℃)	供給流量 (l/m)	供給流れ中の C_2F_6の濃度 (%)	生成物流れ中の C_2F_6の濃度 (%)	C_2F_6の 回収率 (%)
1	25	0.3	2.21	11.74	93.99
2	5	0.3	3.05	13.35	98.95
3	5	0.5	9.76	49.00	87.29
4	25	0.25	5.09	22.93	93.39
5	25	0.50	1.31	5.3	98.44

[0038]Although this invention was explained with the specific experiment in a specific

example, it is thought that an example is only mere illustration of this invention, and various change is possible for it. For example, a bed is constituted from a mixture of two sorts or three sorts or more of adsorbent of an above-mentioned type, or two sorts or three sorts or more of adsorbent can be put in order in series, and can also be used. This invention can only be carried out as an adsorptive process or a part of process of others desirable although perfluoro-carbon is collected again as a part of defecation process of a deposition chamber, or etching process. For example, it can use in order to collect the perfluoro-carbon formed between aluminum refining operations in this invention, in order to separate perfluoro-carbon from the gas for laser (lasing gas), or in order to collect coolant gas. The range of this invention is limited by only the claim.

[Translation done.]

*, NOTICES *

1.This document has been translated by computer. So the translation may not reflect the original precisely.
2.**** shows the word which can not be translated.
3.In the drawings, any words are not translated.

DESCRIPTION OF DRAWINGS

[Brief Description of the Drawings]
[Drawing 1]It is a block diagram showing one example of the device for enforcing the method of this invention.
[Drawing 2]It is a block diagram showing another example of the device for enforcing the method of this invention.

[Translation done.]

* NOTICES *

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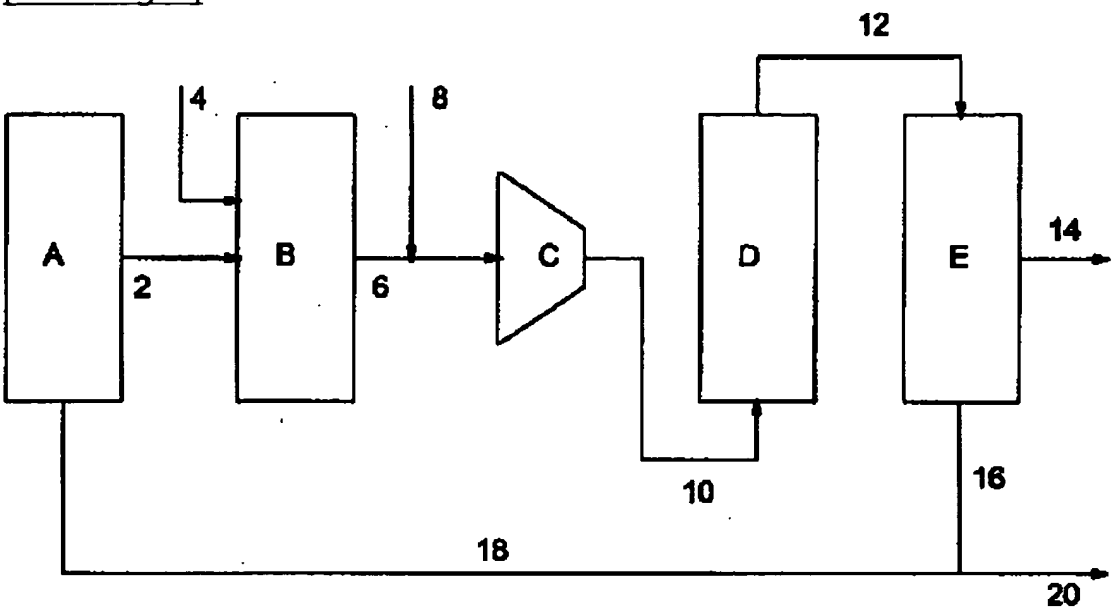
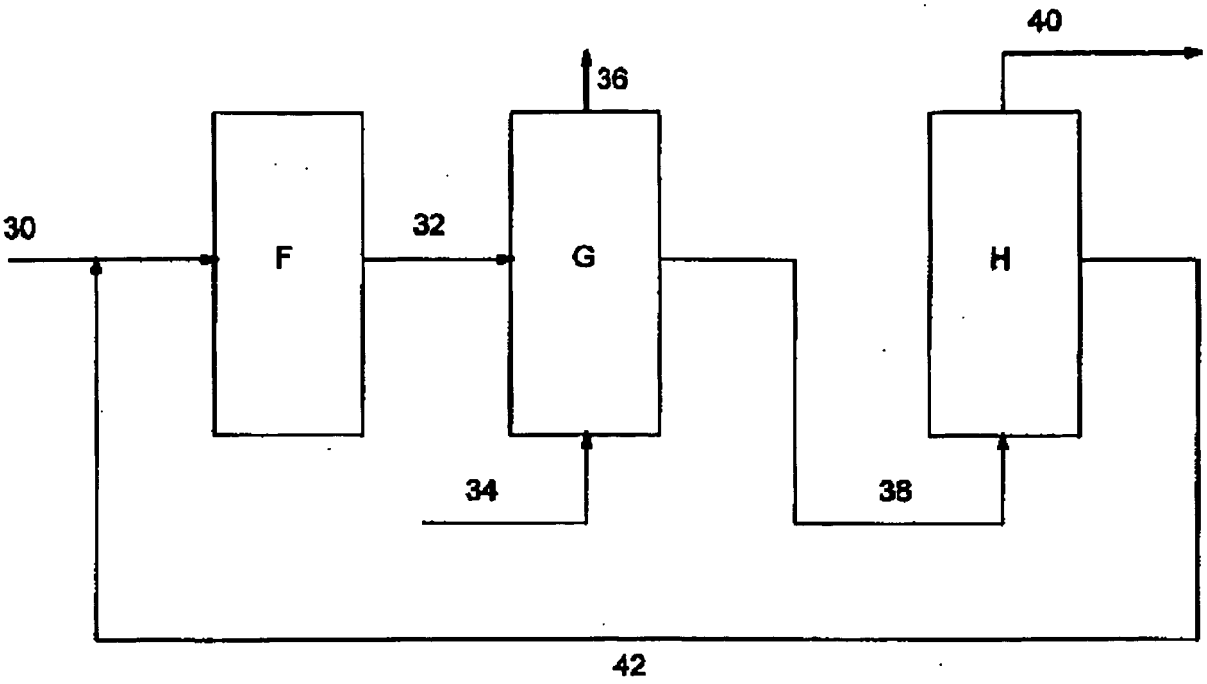
DRAWINGS

[Drawing 1]



[Drawing 2]



[Translation done.]